PATENT SPECIFICATION

Index at acceptance:—Classes 2(2), B1B(1:3); and 120(3), W(1:3).

International Classification:—D01d. (D02d).

COMPLETE SPECIFICATION

Improvements in Melt Spinning Synthetic Polymers

We, E. I. DU PONT DE NEMOURS AND COMPANY, a Corporation organised and existing under the laws of the State of Delaware, United States of America, of Wilmington,
5 State of Delaware, United States of America, do hereby declare this invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the fol-
10 lowing statement: —

This invention relates to the melt spinning of filaments from synthetic polymers.

In the preparation of fibres from fusible polymers, it is customary to force the molten
15 polymer through the orifices of a spinneret into a region where the temperature is lower than the temperature of the molten polymer. In the cooler region, the molten polymer sets into filaments sufficiently firm to be drawn
20 away continuously by a yarn forwarding device. Conventionally, the molten polymer is spun through a spinneret having orifices spaced from each other by relatively large distances in order to keep the newly formed
25 filaments separated until they have congealed sufficiently to prevent their sticking together or coalescing. Productivity of yarn per spinneret under these conditions is low even at the highest practicable speeds of windup.
30 The large hole-to-hole spacing in conventional melt spinning spinnerets contrasts sharply with that used in spinning viscose, where the holes are spaced so closely together that a tow of several thousand filaments can,
35 be spun at a single position. Owing to smaller plant space, smaller investment in equipment, and lower expense of labour and up-keep, this high density of spinneret holes permits the production of fibres at a much lower cost
40 than is possible with conventional melt spinning.

A second disadvantage of known melt spinning practices concerns the difficulty of coupling the steps in yarn preparation. After
45 a yarn is spun, drawing is generally neces-

sary in order to bring its mechanical properties to an acceptable level. However, yarn input to the drawing step usually proceeds at a rate necessarily different from the rate of yarn output from the spinning step. 50
For example, it may be necessary to draw the yarn at a much lower rate than is desirable for spinning it. Under such conditions it is most efficient to interrupt the process of the yarn preparation, that is to package the 55
yarn temporarily after the spinning step for subsequent subjection to the drawing step. Even when it is possible to draw the yarn at a sufficiently rapid rate to allow its being fed directly from the spinning step, the rate of 60
yarn travel at the output from the drawing step often exceeds the capacity of currently available yarn packaging equipment.

This difficulty could be avoided by extruding the yarn slowly enough to allow the drawn 65
yarn to be collected at a reasonable rate. But such an expedient would reduce the productivity of current spinning processes and would, therefore, be economically undesirable. The difficulty could also be avoided if a yarn 70
could be spun which required little or no drawing in order to bring its as-spun properties to an acceptable level.

The present invention comprises a process for the production of filamentary materials by 75
extruding a synthetic molten fibre-forming polymer downwardly through a spinneret having a plurality of orifices, wherein one or more streams of quenching fluid are directed against each filament within one inch of the 80
spinneret face at an angle between 45° below and 45° above the horizontal, the filaments being under a tension of at least 0.003 g/denier and being cooled to a temperature more than 15° C., and preferably more than 25° 85
C. below the melting point of the polymer at a distance less than 2 inches from the spinneret. The invention is of particular value in connection with the use of spinnerets in which the orifices are closely spaced especially by 90

0.125 inch or less, e.g. 0.005—0.125 inch, between centres.

In a preferred embodiment of this invention, the orifices form a pattern in which their centres are positioned at the corners of contiguous quadrilaterals formed by lines connecting the centres of adjacent orifices, each side of each quadrilateral being between 0.005 and 0.125 inch in length, all inside angles in each quadrilateral being at least 30°, and the polymer is extruded at a rate of at least 4 g./min./cm², and preferably at least 10 g./min./cm², of effective spinneret face area within the quadrilateral.

By the expression "effective spinneret face area" is meant the area within a quadrilateral defined by four straight lines between the centres of four adjacent orifices. Thus, if the orifices are arranged in a square pattern, the effective spinneret area is the square of the centre-to-centre spacing of the orifices. When the orifices are arranged in staggered rows, the effective area is a parallelogram with sides corresponding to the centre-to-centre distances in a row and centre-to-centre spacings between orifices in different rows. The manner of determining this value for other arrangements will be obvious.

The invention not only makes it possible to prepare at high productivity yarns which require little or no after-draw, but also affords a particularly advantageous means for melt-spinning filaments having non-circular cross sections. Because of the action of surface tension on the periphery of a freshly extruded and still molten filament, a filament having as extruded a non-circular cross section tends to assume a circular shape before it is congealed or set. In the process of the present invention the surfaces of such filaments are congealed virtually instantaneously, so largely preserving in the filaments the cross-sectional shape of the orifices.

The invention will now be described in greater detail by reference to the accompanying drawings, in which:

Figure 1 illustrates schematically the process of the invention,

Figure 2 shows a spinneret hole pattern suitable for use in the process,

Figures 3 and 4 illustrate a particular apparatus which may be used,

Figures 5—7 show various orifice shapes,

Figures 8 and 9 show the cross sections of filaments spun through the orifices of Figures 5 and 6 respectively, and

Figure 10 shows the cross section of a filament spun through the orifice of Figure 5 without the use of the quenching step of the invention (Example XVIII below).

Referring now to the drawing, Figure 1 shows the invention in its simplest form. Molten polymer is extruded through orifices 1 in spinneret 2, the orifices being arranged in a plurality of parallel straight rows 3 forming a pattern 4 within a perimeter 5. A strong current of quenching gas from nozzle 6 impinges upon the newly formed filaments 7 as they emerge from the spinneret. The bundle of quenched filaments is removed from the quenching region under tension by yarn-forwarding roll 8, which may be either a windup or a guide roll for conveying the yarn to the next processing step, which may be a drawing step. The pattern 4 consists of closely spaced multiple rows of holes 1. The position of the quenching nozzle 6 may be specified by d and h which are, respectively, the vertical and horizontal distances of the nozzle from the centre of the pattern. The orientation of the nozzle is specified by the angle θ, measured from the horizontal, which is the angle the quenching stream makes with the horizontal. Ordinarily, it is advantageous that the quenching stream be imposed perpendicularly to the rows of holes 3 in the pattern 4. However, the quenching stream may be imposed at an angle to the rows of holes. The angle φ is used to designate this angle. The invention is intended to include such a quenching arrangement, so long as the major component of the quenching stream is perpendicular to the rows of holes, that is, so long as φ is less than 45°. The distance s between the rows of holes 3 is no more than 0.125 inch. The pattern 4 need not be rectangular, but may, for example, consist of rows of holes arranged on the circumferences of a plurality of closely spaced concentric circles as shown in Figure 2. For this arrangement of the holes, the quenching stream is preferably radially inward as at S, or radially outward as at B, and the spacing s between circumferences 22 and 23 is less than 0.125 inch.

Figure 3 shows one form of apparatus in greater detail. Molten polymer is forced through sand pack 12 and then through channels 13 leading to holes 14 on the face of the spinneret 15. Band heater 16 may be used to control the temperature of the spinneret independently of the flow of polymer, which heats the spinneret, and of the quenching gas, which cools it. The filaments 17 emerging from the holes are immediately quenched by a strong flow of gas from primary nozzles 18, directed across the bundle of filaments and parallel to the face of the spinneret. In this embodiment, after passing through the initial quenching zone, the filaments enter a secondary cooling zone in which they are subjected to a flow of gas from nozzles 19. This flow of gas further cools the filaments, and also is so imposed as to counteract the deflection due to the action of gas from nozzles 18. Thereafter the filaments pass to a windup or yarn-forwarding device.

Gas supplied to nozzles 18 and 19 passes over flow chopper vanes 20, which serve to distribute it evenly. The nozzles may also

be covered by screens 21 in order to make the flow of gas more homogeneous.

Figure 4 shows in plan the face of one type of spinneret suitable for use with a circular 5″ pack. The working area ABCDEFGH is divided into two parts for more effective quenching. The two areas ABGH and CDEF each contain 810 holes arranged in rows with a centre-to-centre spacing of 0.060 inch between rows and between holes. In each area there are 14 rows of holes perpendicular to the direction of travel of the quenching gas. The position of the quenching nozzles is shown as 18 and 19. Example III below describes in detail the operation of this form of spinneret under a particular set of conditions.

In Figures 5—7 are shown three different non-circular orifices. Obviously, many others are possible, and the only requirement as to shape is that any cross-sectional feature desired to be preserved should be accessible to the quenching stream. That these cross-sectional configurations are preserved in remarkable detail is shown by Figures 8 and 9, which represent actual cross sections of filaments spun from orifices 5 and 6, respectively. The product spun from the orifice of Figure 7 has a triangular cross section. Perfect preservation of shape is not physically attainable, because the effect of surface tension cannot be eliminated completely for any molten material. In the process of the present invention, rounding of both " salient " and " re-entrant " angles is minimised, with the result that important advantages are realised in the fibre.

It is a surprising fact, unrecognised by the art, that multiple rows of holes at close spacing may be employed in conjunction with a strong current of quenching gas, imposed at the spinneret, in the melt spinning of synthetic polymers.

In conventional spinning practice it is not possible to spin a large number of holes at one position. In the case of 3-denier-per-filament polyethylene terephthalate a maximum of about 250 holes can be spun from a 5-inch pack, while in the case of nylon only about 140 holes can be spun. One difficulty which arises when it is attempted to spin more holes is that a stronger current of air must blow across the " chimney " in which the filaments are set. This causes a more severe deflection of the newly formed filaments and leads to yarn which is less uniform. It also causes filaments to move about more, leading to stuck filaments. The fact that the filaments are closer together aggravates the situation and increases the number of stuck filaments still further. Consequently, conventional spinning is very limited in the number of holes which can be spun per position. With the present invention at least 2000 3 denier per filament polyethylene terephthalate or

nylon filaments may be spun from a 5-inch pack.

In the process of the present invention, in which the number of holes in the spinneret and the amount of quenching air are simultaneously increased, the filaments are quickly cooled to a temperature at which their viscosity is sufficiently great to support a much greater tension. Owing to the tension which is imposed upon the threadline at the windup, large deflections of the filaments do not occur. In addition, the length of the critical region where the filaments are tacky is drastically reduced. Both factors reduce the tendency for stuck filaments to appear. In fact, when, in accordance with the invention, the temperature of the threadline at a point two inches below the spinneret is reduced to more than 15° C. and preferably more than 25° C. below the melting temperature, no stuck filaments result.

Thus, in comparison with conventional melt spinning, the present process allows a much larger number of filaments to be spun per spinneret, or alternatively, it allows the same number of filaments to be spun from a much smaller spinneret. Under suitable conditions, it allows a yarn to be spun at a speed which is low enough to allow it to be drawn directly in a coupled process. Alternatively, it allows a yarn to be spun with sufficiently good as-spun properties to render a drawing step unnecessary. In either case the product may be made spontaneously crimpable if desired, by imposing a strong asymmetric quenching stream having a velocity high enough to impart a tension of at least 19 mg./denier to the threadline. By an asymmertic quenching stream is meant a stream which quenches the individual filaments of the yarn at different rates across the yarn. This spontaneously crimpable product has an unusual spiral crimp, and leads to fabrics having greatly improved cover, when compared with fabrics prepared from stuffer-box crimped fibre.

The invention will be further described and illustrated by means of the following Examples.

Examples I and II show that for the polymers polyethylene terephthalate and polyhexamethylene adipamide the invention may be operated under a wide range of conditions, provided the filaments are quenched enough to bring the threadline within two inches of the spinneret to a temperature more than 15° C., and preferably more than 25° C., below the melting point of the polymer.

The expression " relative viscosity " as used herein signifies the ratio of the flow time in a viscometer of a polymer solution containing 8.2%±0.2% by weight of polymer in a solvent to the flow time of the solvent by itself. Relative viscosities are determined at 25° C. Except where otherwise noted, relative

viscosities of polyesters were measured in a mixture of 7 parts by weight tetrachlorophenol and 10 parts by weight phenol, and those of polyamides in 90% formic acid.

5   In all the Examples the threadline tension was greater than 0.003 g./denier.

EXAMPLE I.

Polyethylene terephthalate chip having a relative viscosity of 35 and a melting point of 10 245° C. is vacuum dried for 16 hours at 105° C. The polymer is charged into the hopper of a grid melt spinning unit and blanketed with nitrogen. The temperature of the grid melt unit is maintained at 285° C. The 15 molten polymer is then pumped through a sand pack filter and extruded vertically downward through a spinneret containing 48 holes in a 4 × 12 rectangular array. The diameter of each hole is 0.009 inch, and the centre-to-20 centre spacing is 0.050 inch (0.127 cm.). Thus, the effective spinneret face area is 0.016 cm². The capillary leading to each hole is 0.030 inch long, and is fed through a larger capillary having a diameter of 0.040 inch and 25 a length of 0.35 inch. The spinneret temperature is 275° C.

The newly formed filaments are uniformly quenched with air directed slightly upward and at an angle of 3—4° to the horizontal to-30 ward the centre of the spinneret pattern from a ⅛ inch × 2 inch slot situated 7 inches horizontally and ⅝ inch vertically downward from the face of the spinneret. The long dimension of the slot is parallel to the ground and to the 35 long dimension of the spinneret pattern. Quench air is controlled by means of a Fischer and Porter "Flowrator" (Registered Trade

Mark) (Type B5—27—250/70) with a pressure reducer and pressure gauge upstream of the "Flowrator." Air velocity is measured at 40 the spinneret by means of a Weston or Alnor anemometer.

The temperature of the quenched filaments is measured at a point 2 inches below the face of the spinneret. This measurement is made 45 by a comparison technique, using an infrared vacuum thermocouple as a detector. A strip heater is covered with polyethylene terephthalate film and placed ¼ inch from the filament bundle as a background. A con- 50 cave mirror placed on the opposite side of the bundle focuses infra-red radiation on the detector. As the temperature of the background is raised, the mirror is focussed alternately on the background and on the back- 55 ground and filament bundle together. When the temperature of the background is the same as the temperature of the filament bundle, there will be no change in the output from the detector when the focus is changed. At this 60 point the temperature of the background is determined by thermocouple.

The temperature of the filaments is measured for various throughputs, quench velocities, and windup speeds. The data are 65 presented in Table I. The temperature is observed to vary with the logarithm of the quench velocity.

The tension of the threadline as measured with a tensionmeter 5 feet below the spin- 70 neret for the following spins varied from 3 to 40 milligram (mg.) per denier at 100 yards per minute. Higher tensions accompanied higher quenching rates. Conventional spinning affords tensions of 1 to 3 mg./denier 75 at similar windup speeds.

TABLE I

Filament Temperature Measurement of Jet Quench Spun
Polyethylene Terephthalate Filaments

Run	Throughput (g./min./cm^2)	Denier per Filament	Air Velocity (ft./min.)	Windup Speed y.p.m.	Filament Temp. °C.
1	13.6	10.4	910	100	139
2	13.6	10.4	540	100	189
3	13.6	10.4	380	100	211
4	17.1	27.1	380	100	205
5	17.1	27.1	1900	100	135
6	20.5	32.4	540	100	202
7	20.5	32.4	910	100	176
8	20.5	32.4	1300	100	162
9	20.5	32.4	1900	100	157
10	24.1	38.2	910	100	193
11	24.1	38.2	1300	100	174
12	24.1	38.2	1700	100	165
13	24.1	38.2	2050	100	163
14	17.1	27.1	540	100	198
15	17.1	13.5	540	200	212
16	17.1	6.8	540	400	195
17	17.1	4.5	540	600	187
18	17.1	3.4	540	800	185
19	17.1	2.7	540	1000	182

The above polymer was also extruded from spinnerets having 300 holes spaced 0.018 inch apart on centres, and from spinnerets having 96 holes spaced 0.030 inch apart, using a similar quench.

Operating the above procedure without a quenching fluid at a throughput of from 0.14 to 0.4 g./min./cm^2 product filament temperatures higher than 250° C., and in all cases the filaments were fused together.

EXAMPLE II.

Polyhexamethylene adipamide (nylon) having a relative viscosity of 35 and a melting point of 255° C. is dried, melted and spun by the same procedure as used in Example I.

Threadline temperature is measured in the same way, with the exception that the strip heater is covered with a nylon film to serve as the background. The data are presented in Table II.

The tensions on the threadline vary from 3 to about 60 mg./denier at 100 ypm. In conventional spinning of this polymer at similar speeds and temperature tensions in the range of 1 to 3 mg./denier are observed.

Operating the above procedure without a quenching fluid at a throughput of from 0.14 to 0.4 g./min./cm^2 produced filament temperatures higher than 250° C. and in all cases the filaments were fused together.

Table II

Filament Temperature Measurement of Jet Quench Spun 6—6 Nylon

Run	Throughput (g./min./cm.²)	Denier Per Filament	Air Velocity (ft./min.)	Windup Speed ypm	Filament Temp. °C.
20	14.2	22.6	330	100	209
21	14.2	22.6	540	100	191
22	14.2	22.6	910	100	175
23	14.2	22.6	1300	100	169
24	14.2	22.6	1700	100	171
25	14.2	22.6	2050	100	188
26	14.2	22.6	540	100	191
27	14.2	11.3	540	200	190
28	14.2	5.7	540	400	189
29	14.2	3.8	540	600	187
30	14.2	2.8	540	800	186
31	17.1	27.1	380	100	224
32	17.1	27.1	540	100	210
33	17.1	27.1	910	100	197
34	17.1	27.1	1300	100	202
35	17.1	27.1	1700	100	220

Example III.

Polyethylene terephthalate chip having a relative viscosity of 21.7 is spun from the apparatus shown in Figures 3 and 4. The spinneret holes are 0.007 inch in diameter and have capillaries 0.012 inch in length. Individual counterbores 0.040 inch in diameter and 0.30 inch in length feed the capillaries. The effective spinneret face area is 0.0232 cm². The polymer temperature is maintained at 290° C. The volume of room temperature air delivered from each primary quench nozzle is 60 standard cubic feet per minute (scfm) flowing at an average velocity of 1525 feet per minute. The primary air stream extends from the spinneret face to about 1 inch below the face. The volume of air delivered from each of the secondary nozzles is 18 scfm, flowing at an average velocity of 450 feet per minute. The top of the secondary air stream is located about 1 inch below the spinneret face. Polymer is extruded at a rate of 0.45 gram per minute per hole (19.4 g./min./cm²) and is wound up as a 17.7 denier per filament undrawn yarn at 250 yards per minute. Since there are 1620 holes in the spinneret, this corresponds to a productivity of 96 pounds per hour of 28,700 denier yarn for the entire spinneret. The as-spun yarn has a tenacity of 0.76 g.p.d. and an elongation to break of 406%. The yarn is drawn to 4.6 times its original length (4.6X). Boiling the yarn in water to relax it causes the development of 8 helical crimps per inch. The drawn and relaxed product (4.6 dpf) has a tenacity of 2.9 grams per denier, an elongation of 33%, and an initial modulus of 26.4 grams per denier.

Under appropriate spinning conditions, the rapid quenching which is achieved in the present invention leads to filaments having high as-spun tensile properties. Molecular orientation is induced both during flow of the molten polymer through the channel lead-

ing to the spinneret hole and by stretching as the filament is drawn away under relatively high tension. By the rapid quenching characteristics of the invention, this orientation may be frozen into the polymer structure, and a stronger, stiffer fibre results. When the quenching is carried out asymmetrically, the orientation is preserved to a greater extent on the quenched than on the unquenched side of the filament, and a self-crimpable yarn results. These effects are illustrated in Examples IV, V and X.

EXAMPLE IV.

Polyhexamethylene adipamide having a relative viscosity of 41 is charged into a grid melt spinning unit at 289° C. The polymer is extruded through a spinneret whose holes are set at a 0.050 inch spacing and arranged in a 4 x 12 rectangular pattern. The spinneret temperature is adjusted to 260° C. by means of an auxiliary heater. The polymer is extruded at a rate of 11.0 grams per minute (14.2 g./min./cm²) and wound up at 95 yards per minute. Quenching air is delivered from a Bunsen burner wing tip at a velocity at the spinneret face of 11,000 ft./min. The wing tip is oriented so that its long dimension is parallel to the face of the spinneret and to the long dimension of the hole pattern. It is situated one inch below and 2½ inches horizontally from the centre of the spinneret, and is directed upward at an angle of 17° with the horizontal. The tension on the filament bundle is 22 to 24 grams (19 mg./denier). The properties of the yarn after boil-off are: denier per filament 24; tenacity 0.9 gram per denier; elongation 145%; initial modulus 5.4 grams per denier; 6 to 7 crimps per inch; crimp elongation 185%.

Yarn of this character is directly suitable for use in carpets. In this end use low toughness (i.e. low work to break) is considered advantageous and the carpets do not fuzz or pill. A similar denier as-spun fibre prepared by extruding the above polymer with a conventional chimney quench has a tenacity of 0.8 g.p.d. and an elongation of 450%. Carpets prepared from it fuzz and pill badly.

EXAMPLE V.

Polyethylene terephthalate having a relative viscosity of 34 is charged into a grid melt unit at a temperature of 285° C. and extruded through a spinneret at a temperature of 270° C. The spinneret contains 100 holes at 0.050 inch centre-to-centre spacing arranged in a rectangular 5 x 20 pattern. Polymer is extruded at 10.0 grams per minute (6.2 g./min./cm²) and wound up at 1000 yards per minute. Quenching air is directed from a 1 x 5 inches slot to deliver air at 750 feet per minute at the spinneret. The slot is oriented with its long dimension parallel to the face of the spinneret and to the long

dimension of the hole pattern. It is situated one inch vertically downward from the face of the spinneret and two inches horizontally from the filament bundle. The quenching stream is directed upward at an angle of 30° from the horizontal. As-spun properties are: denier per filament 1.0; tenacity 2.7 grams per denier; elongation 200%; initial modulus 25 grams per denier. The yarn is drawn 2X in water at 90° C. and then has the following properties: denier per filament 0.48; tenacity 5.2 grams per denier; elongation 23%; initial modulus 88 grams per denier. By the process of this invention it is thus commercially feasible to produce fine filaments having a denier from 0.1 to 1.0. This has not been possible heretofore.

The as-spun yarn obtained in Example V, after being boiled off taut, has properties adequate for many end uses.

A further advantage of the present invention is that polymers which would ordinarily be considered unspinnable because of their relatively low melt viscosities can be spun without difficulty. This comes about because the temperature of the extruded polymer is quickly reduced and its viscosity thereby raised to a value at which the filament resists threadline breakage. This circumstance can be put to use in avoiding filtration difficulties. Thus, at the high throughputs achieved in the present invention sufficiently rapid filtration of a polymer melt sometimes presents a problem. For polymers whose fibre properties are not highly sensitive to changes in molecular weight, the problem may be avoided by lowering the molecular weight of the polymer, with a consequent reduction in viscosity and more rapid filtration.

The following Example illustrates the spinning of a polymer not spinnable by conventional means.

EXAMPLE VI.

A sample of polyhexamethylene adipamide having a relative viscosity of 16.5 is spun from a grid melt unit in which the melt is maintained at 270° C. through a spinneret maintained at 250° C. The spinneret contains forty 0.009 inch diameter orifices set at 0.050 inch spacing in a 4 x 10 rectangular array. Quenching air is directed toward the centre of the hole pattern from a ⅛ X 2 inch slot situated 2 inches horizontally from the filaments and one inch vertically downward from the face of the spinneret. The long dimension of the jet slot is parallel to the long dimension of the hole pattern. At a polymer throughput of 0.0815 grams/minute/hole (5 g./min./cm²) and an air velocity at the spinneret of 14,500 ft./min., the yarn is wound up at 95 yards per minute. After being drawn about 4½X on a hot plate at 200° C., followed by a ½ hour boil-off, the yarn

has the following properties: tenacity 5.1 g.p.d.; elongation 31%; initial modulus 22%; denier per filament 2.2. This polymer could not be spun by conventional commercial spinning methods.

Using a similar technique, polyethylene terephthalate with a relative viscosity of 10 is satisfactorily spun.

Examples VII and VIII illustrate the operation of the process with a variety of polymers.

The lowest molecular weight polyhexamethylene adipamide that is commercially spun and drawn at the present time as single component filaments is that which corresponds to a relative viscosity of 27 but, for the production of first-class yarn, relative viscosities of 36 and higher are now used. The commercially acceptable molecular weight levels of other polyamides will vary with the specific polymer, but in general they will be of a magnitude comparable to the above. In contrast, using the process of this invention polyhexamethylene adipamide and similar polyamides having a relative viscosity of at least 12, e.g. in the range of 12 to 20, and of course higher, can readily be spun.

Poly(ethylene terephalate) of relative viscosity 22 or greater must be used in conventional procedures for commercial spinning and drawing, but relative viscosities of 25—33 are currently used in commerce to avoid denier non-uniformities and spinning and drawing breaks, which are prevalent when using the lower molecular weights. Using the process of this invention, such polyesters with a viscosity down to 9 can readily be spun at commercially feasible rates.

EXAMPLE VII.

A sample of "Lustrex-15" ("Lustex" is a Registered Trade Mark) polystyrene flake having a molecular weight of 35,000 to 40,000 and an inherent viscosity of 0.66 at a concentration of 0.5% in benzene, is dried in a vacuum oven at 100° C. and 1 mm. pressure. The polymer is spun from a melt pool at a temperature of 240° C. through a spinneret containing 100 holes arranged in a 10 x 10 square matrix at 0.050 inch spacing. The orifice diameter is .009 inch. The spinneret temperature is varied between 180 and 220° C. The quenching air is directed upwardly at an angle of 18½° with the horizontal. The quenching device is a tube having an inside diameter of ⅜ inch whose orifice is situated 1½ inches below the spinneret and 2 inches from the threadline. The rate of flow of the quenching air is varied between 1.2 and 5.0 scfm. Typical operable spinning conditions are shown in Table III.

TABLE III

Flow of Quenching Air (scfm)	Throughput g./min./cm.^{2}	Windup Speed (yds./min.)	Tension (gms.)	Spinneret Temp. (°C.)
1.2	8.1	60	17	227
2.2	8.1	111	29	220
3.4	8.1	122	65—70	215
5.0	8.1	100	80—90	208
	8.1	25	225	180
1.2	4.7	100	20	227

Tensions on the spinning threadline which are recorded in this Table are exceptionally high even for the present process. The sample spun at a throughput of 8.1 grams/minute/ cm² and at a quench rate of 5.0 scfm has the following properties: tenacity 0.84 g.p.d.; elongation 4%; initial modulus 30 g.p.d.; denier per filament 30. The sample spun at a throughput of 4.7 grams/minute/cm² has the following properties: tenacity 0.94 g.p.d.; elongation 27%; denier per filament 7.7.

EXAMPLE VIII.

An elastomeric copolyester is prepared from ethylene glycol, polytetramethylene oxide glycol having a molecular weight of 1560, and terephthalic acid, the weight ratio of ethylene terephthalate units to polytetramethylene oxide terephthalate units being 2 to 3. The polymer has an inherent viscosity of 1.05 in tetrachloroethane/phenol (66/100). It is stabilised with ½% of an anti-oxidant such as bis(2-methyl-4,6 dihydroxyphenyl)-

methane. The polymer is spun from a grid melt unit maintained at a temperature of 225—245° C. The spinneret temperature is 218° C. The quenching conditions and the spinneret used are the same as those of Example VII. The polymer is spun at a delivery rate (throughput) of 0.075 gram/minute/hole (4.7 g./min./cm²), is quenched with a flow of air at the rate of 1.2 scfm, and is wound up at 35 yards per minute. In order to minimise the tendency of the filaments to stick together, talc is applied to the spinning threadline by means of a flock gun. Properties of the spun yarn are as follows: tenacity 0.15 g.p.d.; elongation 1016%; initial modulus 0.33 g.p.d.; denier per filament 38.2.

EXAMPLE IX.
Semi-dull polyethylene terephthalate hav-

ing a relative viscosity of 34 is spun from a grid melt unit through a spinneret have one hundred 0.009 inch diameter holes arranged in 20 x 5 rectangular array on a 0.050 inch spacing. The threadline is quenched by hot air emerging from a 1¼ x 5 inch slot situated two inches horizontally from the centre of the pattern and one inch vertically downward from the face of the spinneret. The air is directed upward at an angle of 30° with the horizontal. The slot is so oriented that its long dimension is parallel to the long dimension of the spinneret pattern. The velocity of the quenching air at the spinneret is 250 ft./min. Maximum windup rates achieved at two different throughputs for various temperatures of the quenching air are shown in Table IV.

TABLE IV

		Maximum windup speed (yds./min.) at a quench temperature of:		
(gms./min./hole)	Throughput g./min./cm.²	25° C.	100° C.	175° C.
0.13	8.1	900	1200	1500
0.18	11.2	1020	1480	1900

EXAMPLE X.
This Example illustrates a preferred process of this invention.

Polyethylene terephthalate chip having a relative viscosity of 31.2 is melted and the melt (at about 290° C.) is extruded (49—51 pounds of polymer per hour) through a spinneret maintained at 278°—300° C. by an auxiliary electric heater around its circumference. The spinneret comprises 900 holes each 0.007″ in diameter arranged on six concentric circles whose radii differ by 0.052 inch each, the smaller circle of which has a radius of 1.437 inches. The holes are located on radii of the circles. Adjacent radii are spaced 1°12′ apart and contain holes spaced on alternate circles so that a staggered pattern is obtained. Thus, the centre-to-centre spacings in a row (circle) vary from about 0.060 inch in the inner circle to about 0.071 inch in the outer circle. The average effective spinneret face area per hole (taken between rows 3 and 4) is 2.2 x 10⁻³cm², giving a throughput of 18.7—19.5 g./min./cm². The extruded filaments are uniformly quenched with room temperature air from an annular quenching nozzle surrounding the circle of filaments comprising a slot 1 inch high located on the inside surface of a cylindrical chamber having an inside diameter of 4⅛ inches. The top of the slot is spaced 1/16 inch below the

spinneret face by a ring of aluminium foil and heavy asbestos cloth. The filaments are wound up at various speeds and the as-spun filaments are combined to a tow of convenient size and drawn through a hot water bath or spray (about 95° C.) to such an extent as to give a yarn of elongation at the break about 10%. The as-drawn filaments develop a high degree of helical crimp immediately upon release of the drawing tension. The amount of crimp is increased by relaxing the fibres at 100—200° C. (preferably 140° C.) for 2 to 20 minutes. The effect of different amounts of quench is shown in Table V. Item a has 11 crimps per inch of crimped length.

The crimp index, used as a measure of the extent of crimp, is determined from the length of a sample of crimped tow hanging under an added load of 0.1 g.p.d. for a period of 2 seconds (length A) and the length of that tow hanging under no added weight after it has relaxed for 15 seconds from the first extension (length B) where

$$\text{crimp index} = \frac{A - B}{A} \times 100.$$

Items a, c and d in Table V are very useful in stable form as filling for pillows.
Attempts to repeat the above spins with the

substitution of a conventional chimney quenching system with a very high air flow (400 cfm) were unsuccessful at throughput levels of 4.0 g./min./cm² and higher. Air velocity was 311 linear ft./min.

It is to be understood that many process variables control the extent of crimp developed or the crimp index. The following discussion will consider the effect of changing only one variable at a time while leaving all others constant. It will be appreciated that there may be interaction between some variables.

Polymer Viscosity — polyethylene terephthalate of relative viscosity from 9 to 50 can be used. The level of viscosity has no major effect on crimp index but higher viscosities do lead to a higher frequency of crimps.

Spinneret Block Temperature (temperature of the polymer melt before passage through the spinneret). For purposes of making crimped filaments, a higher temperature of the melt is usually required in the case of copolyesters than is normally used in conventional spinning.

Spinneret Temperature — the crimp index is reduced as the spinneret temperature is raised.

Hole Spacings — the hole spacings of this invention, i.e. where the centres of adjacent orifices are less than 0.125 inch apart, are satisfactory.

Hole Size or Orifice Size — in general, larger holes give a higher crimp index level at the crimp index levels of 30 and higher. Thus, a 0.014 inch diameter orifice is preferred in the preparation of products such as shown in Table V.

Air Quench — as the air quench is increased, the crimp index is increased. A radially directed inward quenching stream at 0° to horizontal is preferred as producing substantially more uniform products.

Polymer Throughput — as the amount of polymer going through an orifice in a given time increases, the crimp index of the final yarn decreases.

Total Draw Ratio—this should be considered as the total orientation obtained in combination by spinning and drawing, as the spinning conditions will control the absolute draw ratio. However, as the draw ratio is decreased below that required to give a break elongation (on unrelaxed drawn yarn) of less than about 17%, the crimp index decreases rapidly. Preferably, to gain maximum crimp index, the yarn should be drawn to give the unrelaxed drawn yarn a 10% elongation at break.

Drawing Conditions — drawing in a bath or spray of hot water is preferred. Drawing over a hot pin (e.g. at 80—90° C.) lowers the crimp index.

Filament Denier — as the denier is increased, the crimp index is reduced.

EXAMPLE XI.

Polypropylene with a melt index of 0.7 (ASTM D—1238—57T) is extruded at a rate of 5.2 g./min./cm² from a pool of the molten polymer at 280—290° C. through a spinneret maintained at 260° C. and the yarn wound up at 183 y.p.m. The spinneret contains 38 holes 0.009 inch in diameter in 4 rows, the holes being arranged in a square pattern 0.050 inch apart in each direction from adjacent holes. A quenching nozzle with a width of 1.25 inches and a height of 2 inches is placed with its long dimension vertical, ½ inch below the face of the spinneret and 1 inch horizontally from the outer row of filaments. Room temperature air is then fed through this nozzle at a rate of 21 scfm, which affords a tension on the threadline of 28 mg. per denier.

The as-spun yarn (4.7 dpf) is found to have a surprisingly low crystallinity (about 0.6 of the level obtained from this polymer by conventional quenching methods), and after relaxation (boiling in water), has a tenacity of 1.9 g.p.d., a break elongation of 360%, an initial modulus of 7, and 10—15 crimps per inch.

The as-spun yarn is then drawn 1.67X in 96° C. water and then further drawn 1.91X over a metal plate heated to 112° C. to give a total draw of 3.2X. The drawn yarn, after relaxing by boiling in water, has a tenacity of 5.9 g.p.d., an elongation of 30% at the break, and an initial modulus of 29 g.p.d. The filaments are straight and uncrimped.

TABLE V

Item	Windup Speed, y.p.m.	Spinneret Temp., °C.	Quenching Conditions		Relaxed Fibre Properties			
			cu. ft. air/lb. polymer	Total Draw Ratio	Crimp Index	Tenacity g.p.d./Elongation %	Initial Modulus	d.p.f.
a	310	278	245	3.54	51	3.4/25	52	4.6
b	400	300	25	5.0	<5	—/25—30		3.0
c	400	270	93	4.55	32	4.2/32	63	3.8
d	400	282	188	3.33	60	3.7/26	43	3.6

Another advantage of the rapid quench method of this invention is the ability to spin polymer through an orifice having a non-round cross-section to obtain a filament having an arbitrary cross-sectional shape substantially the same as the orifice. This feature is shown in the following Examples, in which all orifice spacings are measured centre-to-centre.

EXAMPLE XII.

Polyhexamethylene adipamide having a relative viscosity of 39 is spun from a melt at a temperature of 288° C. through a spinneret at a temperature of 260° C. This spinneret contains 35 holes arranged in a 7×5 staggered, rectangular pattern, the distance of each hole from its nearest neighbours being 0.050 inch (centre-to-centre). Each hole consists of three slots, 0.010 inch long and 0.003 inch wide, set radially at angles of 120° around a 0.007 inch circular centre hole. The polymer is extruded at a rate of 0.3 gram per minute per hole (18.6 g./min./cm²). The quenching medium is delivered through a cylindrical sintered bronze surface which is 2 inches in inside diameter and 1½ inches long, and is situated coaxially with the yarn bundle with its top ¾ inch below the face of the spinneret. The quenching medium is air, at 70° F. delivered at 14 scfm (standard cubic feet per minute) radially inward toward the yarn bundle.

The yarn is drawn over an 8-inch hot plate. Table VI gives physical properties of the yarn for two sets of operating conditions.

TABLE VI

Speeds (y.p.m.)		Draw Temp.	Denier per Filament	T	E	Mi
Feed Roll	Draw Roll					
93	188	218°C.	15	1.3	168	6.0
45	90	200°C.	35	1.75	225	9.6

T = tenacity (grams per denier)

E = elongation (%)

Mi = initial modulus (grams per denier)

The 35 d.p.f. yarn of this character is especially suitable for use in carpets because of its bulkiness and crispness.

When the same spinning procedure is followed but with conventional quenching as described in United States Patent No. 2,273,105, the yarn produced has a maximum drawn denier of 17, since it must be wound up no slower than 100 yards per minute, and the filament cross section is a rounded triangle in contrast to the Y-shaped cross section prepared above.

EXAMPLE XIII.

Polyhexamethylene adipamide nylon polymer having a relative viscosity of 41 is heated to 292° C. and extruded through a spinneret whose temperature is 270° C. The spinneret comprises 10 holes situated in 2 rows of 5 holes each. The holes form a 60° parallelogram such that each hole is ⅛ inch from adjacent holes. The holes have the modified triangular cross section shown in Figure 7, where the sides of the central equilateral triangle are 0.015 inch long, and the three rectangular slots are 0.006 inch long and 0.003 inch wide. The filaments are first quenched by a jet of air from a 6 × 1½ inch slot situated such that its long dimension is flush with the spinneret. An additional quench is supplied at 7.3 scfm from a ½ inch wide slot on a cylinder 2 inches in diameter positioned co-axially with the yarn bundle at a distance of 1¾ inches vertically downward from the face of the spinneret. The primary quenching stream is directed upward at the centre of the spinneret pattern at an angle of 45° with the horizontal. The throughput is at a rate of 3.3 grams/minute/hole (37.8 g./min./cm²), and the yarn is wound up at a rate of 1000 yards per minute. The fibres composing the yarn bundle are very nearly triangular in cross section and have the following as-spun properties: tenacity 1.0 gram/denier; elongation 375%; initial modulus 6 to 8 g.p.d.; denier/filament 33. Yarns of this description may be used directly in the preparation of rug piles having exceptional bulk, a luxurious hand, and improved crush resistance.

EXAMPLE XIV.

Polyhexamethylene adipamide having a relative viscosity of 41 is extruded from a melt pool at a temperature of 288° C. through a spinneret at a temperature of 275° C. The spinneret comprises 48 holes arranged in 6 rows of 8 holes each, alternate rows being staggered so that the centre of each hole is at the vertex of an equilateral triangle of side 0.050 inch. The shape of the orifices is shown in Figure 6. The quenching air is supplied from a Bunsen burner wing-tip situated 1½ inches below the spinneret and 4 inches horizontally from the centre of the spinneret pattern and directed upward toward the centre of the pattern. The properties of the product are given in the following Table. The running tension on the yarn bundle, as measured on a tensometer, is given in grams per denier.

TABLE VII

Throughput (gm./min./ cm²)	Quench Rate (scfm)	Running Tension (gm./den.)	Windup Rate (yd./min.)	T	E	Mi	D
14.7	4.2	0.29	95	0.7	127	3.1	16.5
3.7	1.6	0.069	200	1.7	103	4.8	2.6

T   = tenacity (grams per denier)

E   = elongation (%)

Mi = initial modulus (grams per denier)

D = denier per filament

The first item in the above Table (95 yards per minute windup rate) can be processed directly into an excellent carpet that does not pill or fuzz. As-spun filaments made by conventional spinning procedures using the same orifices have rounded triangular cross sections, a tenacity of 0.8 g.p.d., and an elongation of 450%. Carpets made from yarns of such filaments do pill and fuzz.

EXAMPLE XV.

Polyethylene terephthalate having a relative viscosity of 34 is extruded from a melt at a temperature of 281° C. through a spinneret at a temperature of 288° C. The spinneret comprises 32 holes arranged in straight rows such that the centres of the holes are at the vertices of equilateral triangles whose sides are 0.050 inch. The holes have the shape shown in Figure 5. The extruded filaments pass through a cylindrical quenching zone extending from $\frac{1}{4}$ inch to $1\frac{3}{4}$ inch below the face of the spinneret and oriented coaxially with the filament bundle. The quenching medium is air, delivered through the face of a sintered bronze tube whose inside diameter is $1\frac{3}{8}$ inches. The quenching air is supplied at a rate of 7.3 scfm. The quenched filaments are wound up at a point 4 feet below the quenching zone at a rate of 1100 yards per minute. The throughput is at the rate of 13 grams/min./cm². The filaments have the cross-sectional shape of Figure 8, showing excellent retention of the orifice shape.

EXAMPLE XVI.

Polyethylene terephthalate having a relative viscosity of 35 and containing 0.3% TiO₂ is extruded from a melt at 286° C. through a spinneret maintained at 274° C. The spinneret comprises 33 holes arranged in 5 rows so that the centre of each orifice is 0.07 inch from the centres of the adjacent orifices. The orifices consist of 6 rectangular slots, each 0.003 inch by 0.012 inch long, radiating from a common centre point at 60° angles to comprise a six-membered star. Polymer passing through each orifice at the rate of 0.43 gram per minute (13.6 g./min./cm²) is quenched by air at 75° F. from a circular quenching apparatus directed inward at the threadline at a value of 5.5 scfm (70° F. 1 atmosphere, absolute). The quenching apparatus consists of a porous bronze tube of 1.75 inches I.D. and 1.5 inches long located about 0.25 inch below the face of the spinneret. The yarn is wound up at 1230 yards per minute and is 115 denier as spun.

The as-spun yarn is drawn over a hot pin at 110° C. at 100 yards per minute to give 70 denier yarn (1.65X draw ratio) with the following properties: tenacity 3.58 g.p.d.; elongation 66%; and modulus 51 g.p.d. The drawn yarn was then made into a taffeta fabric of plain weave with a finished construction of 128 x 80 having a finished weight of 2.10 ounces per square yard, a bulk of 2.56 cm³/gram, and a coefficient of friction (fabric on fabric) of 0.75. This yarn offers new and novel fabrics that are especially valuable as sheeting.

EXAMPLE XVII.

Polyhexamethylene adipamide with a relative viscosity of 34 and containing 0.3% of TiO₂ as a delusterant is extruded from a melt at a temperature of 287° C. through a spinneret at a temperature of 274° C. Each orifice of the spinneret consists of a 0.007 inch centre hole with 6 slots 0.006 inch long by 0.003 inch wide radiating outward at 60° angles from the circumference of the centre hole (see Figure 5). Thirty-two of the orifices are arranged in 5 rows with 6 and 7 orifices in alternate rows and orifices in adjacent rows being staggered. The orifices are located 0.050 inch apart on centres in a row and the rows are spaced 0.050 inch apart. Room tem-

perature air at a rate of 1.5 scfm is supplied through a ½ inch by 3 inch slot directed upwards at an angle of 14° to the horizontal to hit the threadline about 0.25 inch below the spinneret face. The quenching device is located one inch below the face of the spinneret and three inches away from the spinneret centre line. The polymer is extruded at a rate of 0.208 gram per minute (12.9 g./min./cm²) and the yarn is collected and drawn over a hot plate, the feed rolls running at 620 yards per minute and the draw rolls operating at 950 yards per minute to give a draw ratio of 1.53X. The as-drawn has a tenacity of 2.6 g.p.d., an elongation of 84%, and an initial modulus of 11.7 g.p.d.

The as-drawn yarn was then made into a taffeta fabric of plain weave with a finished fabric construction of 135 × 81 having a finished weight of 2.1 ounces per square yard, a bulk of 2.72 cm³/gram, and a coefficient of friction (fabric on fabric) of 0.66.

EXAMPLE XVIII.

This Example shows the preparation of a product outside the scope of this invention for comparative purposes. The polymer of Example XVII is extruded at 278° C. through a spinneret maintained at 277—280° C. similar to that used in Example XVII except each of the 6 slots are 0.003 by 0.018 inch in dimensions. The orifices are arranged on a 1.25 inch diameter circle. Two rows of orifices are located on parallel chords each 11/64" from the centre of the circle. Two other rows containing 6 and 7 orifices are located on the circumference of the circle so that the chord joining the ends of the row is approximately parallel to the 2 straight rows. All orifices are 0.050 inch apart in a row (centre to centre). The polymer is extruded at a rate of 0.41 gram per minute per hole (1.5 gm./min./cm²). No quenching is employed. The yarn is wound up at 550 yards per minute and drawn 3.6X over a 100° pin to give a yarn with a tenacity of 4.80 grams per denier, an elongation of 36%, and an initial modulus of 85.

A taffeta fabric of plain weave is prepared from the as-drawn yarn to give a finished construction of 117 × 90 and a fabric weight of 2.18 ounces per square yard, a bulk of 1.76 cm³/g., and a coefficient of friction (fabric on fabric) of 0.35.

The coefficient of friction is measured between 2 fabrics mounted on a 45° plane and a sliding block so that both fabric fillings are parallel with the direction of moving. The force required to cause the block to move at a slow steady speed is used in the calculation.

The fabric bulk is calculated from weight per square yard and fabric thickness measured by ASTM procedure D—39—49 with thickness gauge ASTM D—76—53.

The increased bulk and the much higher friction of the items in Examples XVI and XVII, compared with Example XVIII, were quite surprising inasmuch as the only different process variable was the use of the spinning conditions of this invention.

Figures 8 and 10 show the cross sections of filaments obtained by Example XVI and this Example, respectively.

Many useful products, which were heretofore unachievable, may be prepared using the process described above. The prismatic filaments of this invention are distinguished over previous filaments of non-circular cross section by the well-defined angles between sides.

Filaments so characterised give fabrics which have higher bulk, greater resilience, and better handle than previous non-round filaments. Because of the sharper edges, filament-to-filament contacts tend to be more firmly made, and filaments do not slide or roll over one another. Thus, when a fabric is compressed, its structure recovers in spring-like fashion upon release of pressure. This is a factor in the improved crush resistance of carpets prepared from such filaments.

Garments prepared from these filaments are more comfortable to wear. There is less filament surface in contact with the skin. Grooves formed by the re-entrant angles allow ventilation between the fibre and the skin and also improve wicking action, facilitating removal of moisture from the skin.

EXAMPLE XIX.

Polypropylene with a melt index of 0.7 (ASTM D—1238—57T) is extruded at a rate of 6.2 g./min./cm² from a pool of molten polymer at 280—290° C. through the spinneret of Example XVII maintained at 260° C. and the yarn is wound up at 170 yards per minute. A quenching nozzle with a width of 1.25 inches and a height of 2 inches is placed with its long dimension vertical, ½ inch below the face of the spinneret and 1 inch horizontally distant from the outer row of filaments. Room temperature air is then fed in a horizontal direction through this nozzle at a rate of 15 scfm, which affords a tension on the threadline of 30 mg. per denier.

The as-spun yarn (4.9 dpf) is found to have a cross section very similar to Figure 8 but more closely resembling the orifice shape. This is very unusual especially at such low deniers. After relaxation by boiling in water, the as-spun yarn has a tenacity of 2.0 g.p.d. with a break elongation of 350%, an initial modulus of 7, and is crimped. The yarn can be drawn to increase its strength to 6 g.p.d. or higher.
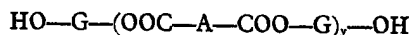
By the term "arbitrary cross section" is meant a cross section of a filament which has a perimeter substantially larger than the circumference of a round filament of the same denier. In other words, "arbitrary cross

section" means odd or non-round cross section.

The invention is applicable to any melt-spinnable man-made organic thermoplastic fibre-forming polymer. A preferred species of this group is the crystallisable polymers. Examples of suitable synthetic polymer are polyamides, polyesters, polyhydrocarbons such as polypropylene, polyurethanes, polyureas, vinyl polymers such as polyvinyl chloride, polyvinylidene chloride, and copolymers thereof, acrylic polymers such as polyacrylonitrile when sufficiently plasticised to render it fusible, copolymers of acrylonitrile, halogenated hydrocarbons such as polychlorotrifluoroethylene, polyacetals, polyanhydrides, polyoxymethylenes, polyformals, polyethers, polythioethers, polysulphides, polythioesters, polysulphones, polythioureas, polythioamides, polysulphonamides, polyimides, and polytriazoles. Copolymers of all sorts are usable. Of course, a small amount of plasticiser may be present in the resin.

Because of their commerical availability, ease of processing and excellent properties, the condensation polymers and copolymers, e.g. polyamides, polysulphonamides and polyesters, and particularly those that can be readily melt spun, are preferred for application in this method. Suitable polymers can be found for instance among the fibre-forming polyamides and polyesters which are described, for example, in Specifications Nos. 461,236 and 578,079 and United States Patents Nos. 2,071,250; 2,130,523; 2,130,948; and 2,190,770. The preferred group of polyamides includes homo polymers and copolymers of hexamethylene adipamide, hexamethylene sebacamide and epsilon-caproamide.

The preferred group of polyesters are linear terephthalate polyesters. These should be fibre-forming and have a relative viscosity of at least 9, preferably at least 12. Such polymers may be represented in a more general way by the formula

$$HO—G—(OOC—A—COO—G)_y—OH$$

where —G— and —A— are divalent organic radicals corresponding, respectively, to the radicals in the initial glycol, $G(OH)_2$, and to the initial dicarboxylic acid, $A(COOH)_2$, and y is a number sufficient for the polymer to have a fibre-forming molecular weight, at least about 75% of the —A— radicals being terephthalate radicals. Preferably, at least about 75% of the —G— radicals are ethylene radicals. The terephthalate radical may be the sole dicarboxylate constituent of the recurring structural units, or up to about 25% of the recurring structural units may contain other dicarboxylic radicals, such as the adipate, sebacate, isophthalate, 5-(sodium sulpho)isophthalate, bibenzoate, hexahydro-terephthalate, diphenoxyethane - 4,4¹ - dicarboxylate, or p,p^1 - sulphonylbibenzoate radicals, derived from the corresponding dicarboxylic acids or ester-forming derivatives thereof. Similarly, ethylene glycol may be the sole glycol constituent of the polyester, or another glycol may be used, such as tetramethylene glycol, hexamethylene glycol, decamethylene glycol, 2,2 - dimethylpropanediol, trans - p - hexahydroxylylene glycol, diethylene glycol, bis - p - (β-hydroxyethoxy)-benzene, bis - 1,4 - (β - hydroxyethoxy) - 2,5-dichlorobenzene, or bis - [p - (β - hydroxyethoxy)phenyl] - difluoromethane alone or in mixtures.

The spinneret may have a flat face or a face so sculptured as to facilitate cooling of the vicinity of the orifices, either symmetrically or asymmetrically. For instance, the face of the spinneret may have flanges or grooves in the direction of quenching in order to conduct the flow of gas in a more uniform fashion. If desired, the holes may also be placed on individual nipples in order to facilitate rapid cooling.

Although the nature of the quenching gas influences optimum operating conditions because of its heat conductivity, any gas can be used, e.g. air, nitrogen, helium, carbon dioxide, or water vapour. The quenching stream may contain suspended droplets of fluid, as well as finely divided solid particles of granular, fibrous or plate-like form. These may serve to modify the rate of cooling or crystallisation, or change the surface characteristics of the fibres.

When spinning through non-circular orifices the quenching medium must be supplied in a volume such as to cool the surfaces of the molten filaments to a temperature at which the filament becomes sufficiently solid to resist deformation of the cross section by the force of surface tension. In addition, the quenching medium acts to cool the face of the spinneret and must be supplied in sufficient quantity to accomplish this end. The volume of quenching medium required will depend on the nature of the polymer, the quenching geometry, and the cross section of the fibre which is being quenched. While it is not possible to predict the optimum quantity of quenching medium required under a given set of circumstances, one skilled in the art, after study of the foregoing Examples, will have no difficulty in ascertaining what quantity of quenching medium to use in any instance. A very useful guide in this work is the tension on the spinning threadline, which may be measured with any of the various commercially available tensometers. In general, tensions of more than 0.003 g.p.d. should be used. The quenching medium as used in all processes of this invention should have a velocity of at least 250 ft./minute at the point of contact with the filaments.

The quenching medium should be delivered to the region of the orifices in a homogeneous, evenly distributed stream. It is the practice in conventional melt spinning to place the spinneret in a recess in order to avoid all cooling of the spinneret face by the quenching medium. By contrast, in the present process, it is desirable that a substantial stream of gas strike the face of the spinneret in the region of each orifice. Although quenching of the filaments by a stream of air moving parallel to the face of the spinneret at a distance somewhat below the spinneret will produce filaments of non-circular cross section, to obtain non-round filaments whose cross section closely approximates to the contours of the spinneret orifices it is necessary that the filaments be quenched immediately upon emerging from the orifices, and this is possible only if at least a portion of the quenching stream strikes the spinneret face itself.

The quenching medium may be supplied in any satisfactory manner. For example, gas from a supply source can be delivered to the interior of a hollow circular cylindrical chamber. The gas leaves through the porous inner surface of the chamber, that is, the surface having the smaller diameter. A slotted toroidal plenum chamber may also be used. In this case the gas may be directed upwardly at not more than 45° to the horizontal in a converging path, coaxial with the filament bundle, such that each spinneret hole of the pattern is subjected to approximately the same flow of gas.

Alternatively, the source of the quenching stream may be situated within the conical array formed by the filaments as they converge below the spinneret. In this case, the quenching medium is directed upwardly at not more than 45° to the horizontal and outwardly in order to strike the spinneret in the region of the orifices. If the orifices are arranged in a rectangular pattern, the quenching medium may be directed at the orifices from one or more slots or rows of holes oriented parallel to the sides of the rectangle.

In general, it is not possible to quench each orifice in a completely uniform manner if there are many rows of filaments. This is readily apparent, inasmuch as orifices which are on the interior of the pattern are quenched by gas which has traversed one or more rows of hot filaments. Consequently, the temperature will be somewhat higher than for the case of the orifices on the periphery of the pattern. Therefore, when a highly uniform yarn is desired, it is advantageous to quench not more than about 4 rows of filaments, when quenching is unilateral.

It is especially difficult by conventional means to spin high denier filaments with non-round cross sections, particularly at high productivity. In the present process filaments having as-spun deniers up to 150 can be prepared. Drawn yarns having deniers per filament of 15 to 35 can easily be spun at a rate of 3.5 grams/minute/hole. These yarns show surprisingly good dye takeup and dyeing uniformity.

As previously indicated the type of quenching fluid used is not critical. However, the use of steam, either wet or dry, offers special advantages in that a more uniform yarn bundle is produced, as evidenced by the increased tenacity obtained upon drawing. In general, the greater the draw ratio, the higher the tenacity. Since the maximum draw ratio which can be achieved depends upon the maximum draw ratio of the least drawable filament, uniformity of filaments in a bundle is critical to obtain the high tenacity yarns.

The following Example demonstrates that the favourable results achieved by quenching with dry steam are not duplicated when other gases are used. Example XXI illustrates the use of wet steam.

EXAMPLE XX.

Polyethylene terephthalate with a relative viscosity of 32 is extruded from a melt at a temperature of 290° C. through a horizontal spinneret having 126 0.007-inch diameter holes arranged in a 7 x 18 rectangular pattern at a centre-to-centre distance of 0.050 inch. Polymer is extruded at a rate of 0.2 gram/min./hole. The quenching device is a flared tube 15 inches long, which receives the quenching medium from a $\frac{1}{4}$ inch internal diameter pipe and discharges it from a nozzle having dimensions 1 x 2 inches. The nozzle is oriented with its long dimension parallel to the long dimension of the spinneret pattern, and is situated so that the centre of the orifice is 1 inch vertically distant from the face of the spinneret and $1\frac{1}{2}$ inches horizontally distant from the centre of the spinning threadline. It is tilted upward so that the quenching stream is at an angle of 20° with the horizontal. Steam is directed at the filaments at a rate of 1—2 pounds per pound of polymer. The quenched filaments are wound up at a speed of 1000 yards per minute as about 2 denier per filament as-spun yarn. The yarn is subsequently drawn at 90° C. over a hot pin at the maximum operable draw ratio. Yarn elongation is measured on the undrawn yarn. Maximum draw ratio and elongation are measures of yarn uniformity. In the following Table, the values of these properties are compared as functions of the nature and temperature of the quenching stream. The extrusion rate of 0.2 g./min./hole is eqivalent to 12.4 g./min./cm² of effective spinneret face surface area.

TABLE VIII

Quenching Medium	Quenching Temperature (°C.)	Quenching Rate (lb./min.)	Draw Ratio	Elongation (%)
Air	35	.24	2.6	225
	77	.24	2.4	215
	140	.24	2.7	232
Steam	105	.25	3.4	328
	125	.25	3.3	400
	140	.25	3.4	366
	185	.25	3.0	339
Carbon dioxide	144	.32	2.6	—

EXAMPLE XXI.

Polyethylene terephthalate having a relative viscosity of 31.6 is extruded at a temperature of 290° C. from a 126-hole spinneret. The spinneret holes have a diameter of .0050 inch and are situated in a rectangular array 18 holes long by 7 holes wide, with a centre-to-centre separation of .050 inch. The filaments are quenched with wet steam which is delivered from a rectangular slot 1½ inches high 3 inches wide at an upward angle of 15 to 20° with the horizontal at a rate of 1—2 pounds per pound of polymer. The slot is 2 inches distant from the threadline and one inch vertically downward from the spinneret, with its long dimension parallel to the long dimension of the spinneret pattern. The quenched filaments are wound up at speeds of 475 and 1050 yards per minute. The properties of the as-spun yarn are given in Table IX. Properties of the same yarns, after drawing over a hot pin at a temperature of 90° C., are also give in the Table. Wet steam refers to steam at 100° C. and under atmospheric pressure.

TABLE IX

Windup Speed (yds./min.)	As-Spun Props.				Draw Ratio	Props. After Draw			
	T	E	Mi	D		T	E	Mi	D
475	1.4	390	35	2.9	4.9	5.5	17	97	.64
1050	1.9	330	38	1.5	2.7	4.1	59	68	.60
1050	1.7	295	29	2.7	2.8	4.8	41	84	.83
1050	1.5	370	31	4.0	3.1	4.1	41	69	1.5

T is the tenacity in grams/denier, E is the elongation in %, Mi is the initial modulus in grams/denier and D is the denier per filament.

Under comparable conditions, yarn which is spun at 1000 yards per minute and quenched with air at 23° C. instead of steam has a maximum draw ratio of only 2.2 to 2.3. In the above Table polymer throughput in g./min./cm² of spinneret face surface area is 8.7, 10.0, 18.0, and 26.6, respectively, reading from top to bottom.

The quenching geometry is not critical. However, it is important that the quenching zone should be as close to the spinneret as possible. The quenching medium may be imposed, for example, unilaterally, bilaterally, radially inward, and radially outward. The length of the quenching zone will be a function of the rate at which polymer traverses the zone as well as of the temperature to which the polymer is to be quenched.

It is sometimes desirable that the yarn be allowed to approach room temperature before being collected. This is easily accomplished by allowing the yarn a few feet of free travel from the point at which it leaves the quenching zone to the point at which it is wound up. However, if space is an important consideration, the yarn may be collected immediately after it leaves the quenching zone. Since the quenching zone itself may be very short, there is a saving of space when compared with conventional spinning.

In the case of wet steam it is very surprising that the presence of liquid droplets in the quenching stream should lead to a yarn of higher uniformity. In fact, quite the reverse effect would be expected, inasmuch as liquid droplets striking the filaments would be expected to cause strong local quenching, leading to non-uniformity in the filaments. However, in the operation of the present invention with wet steam, the droplet diameter is maintained at less than half the diameter of the unquenched unextended filaments, and preferably less than a tenth of that diameter, so that the droplets are apparently either completely vaporized or are deflected before striking a filament and causing a non-uniform section. A beneficial effect of the presence of the droplets is the heat balance in the quenching medium which is achieved in their presence. Heat which is removed from the filament bundle during the quenching process acts to evaporate liquid from the surface of the droplets. As the quenching medium traverses the filament bundle there need be no rise in temperature, but only the evaporation of a portion of the droplets.

Thus, when wet steam is used, the present process provides a method for positive temperature control of the spinning environment which heretofore has not been achieved in melt spinning. This temperature control in the quenching region makes possible improved uniformity of the product.

Yarns and tows prepared in accordance with this invention may be converted into woven and non-woven fabrics by methods well known to those skilled in the art.

WHAT WE CLAIM IS:—

1. A process for the production of filamentary materials by extruding a synthetic molten fibre-forming polymer downwardly through a spinneret having a plurality of orifices, wherein one or more streams of quenching fluid are directed against each filament within one inch of the spinneret face at an angle between 45° below and 45° above the horizontal, the filaments being under a tension of at least 0.003 gram per denier and being cooled to a temperature more than 15° C. below the melting point of the polymer at a distance less than 2 inches from the spinneret.

2. Process according to claim 1 wherein the orifices are spaced one from the other by 0.125 inch or less between centres.

3. A process according to claim 2, wherein the orifices from a pattern in which their centres are positioned at the corners of contiguous quadrilaterals formed by lines connecting the centres of adjacent orifices, each side of each quadrilateral being between 0.005 and 0.125 inch in length, all inside angles of each quadrilateral being at least 30°, and the polymer is extruded at a rate of at least 4 g./min./cm² of effective spinneret face area (as hereinbefore defined).

4. A process according to claim 3, wherein the polymer is extruded at a rate of at least 10 g./min./cm² of effective spinneret face area.

5. A process according to any one of claims 1—4, wherein the quenching fluid is air.

6. A process according to any one of claims 1—4, wherein the quenching fluid is dry steam or wet steam.

7. A process according to claim 6, wherein the quenching fluid is wet steam containing water droplets substantially all of which have diameters less than half the diameter of the filaments extruded.

8. A process according to any one of the preceding claims, wherein the quenching fluid travels asymmetrically with respect to the filaments.

9. A process according to claim 8, wherein the stream of quenching fluid has a velocity high enough to impart a tension of at least 19 mg./denier to the filaments, and the filaments formed are subsequently relaxed to develop a crimp therein.

10. A process according to any one of the preceding claims, wherein the orifices are non-circular.

11. A process according to any one of the preceding claims, wherein the polymer is a linear polyamide, polyester, or polyolefin.

12. A process according to claim 11, wherein the polymer is a polyhexamethylene adipamide of relative viscosity 12 or higher.

13. A process according to claim 11, wherein the polymer is a polyethylene terephthalate of relative viscosity 9 or higher.

14. A process according to claim 11, wherein the polymer is a fibre-forming polypropylene.

15. A process according to any of the preceding claims, wherein the spinning conditions

are such that the filaments formed, after a subsequent drawing step, have an individual denier between 0.1 and 1.0.

16. A process for the production of filamentary materials according to claim 1 substantially as hereinbefore described.
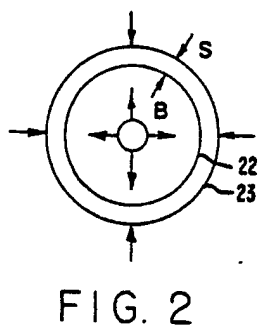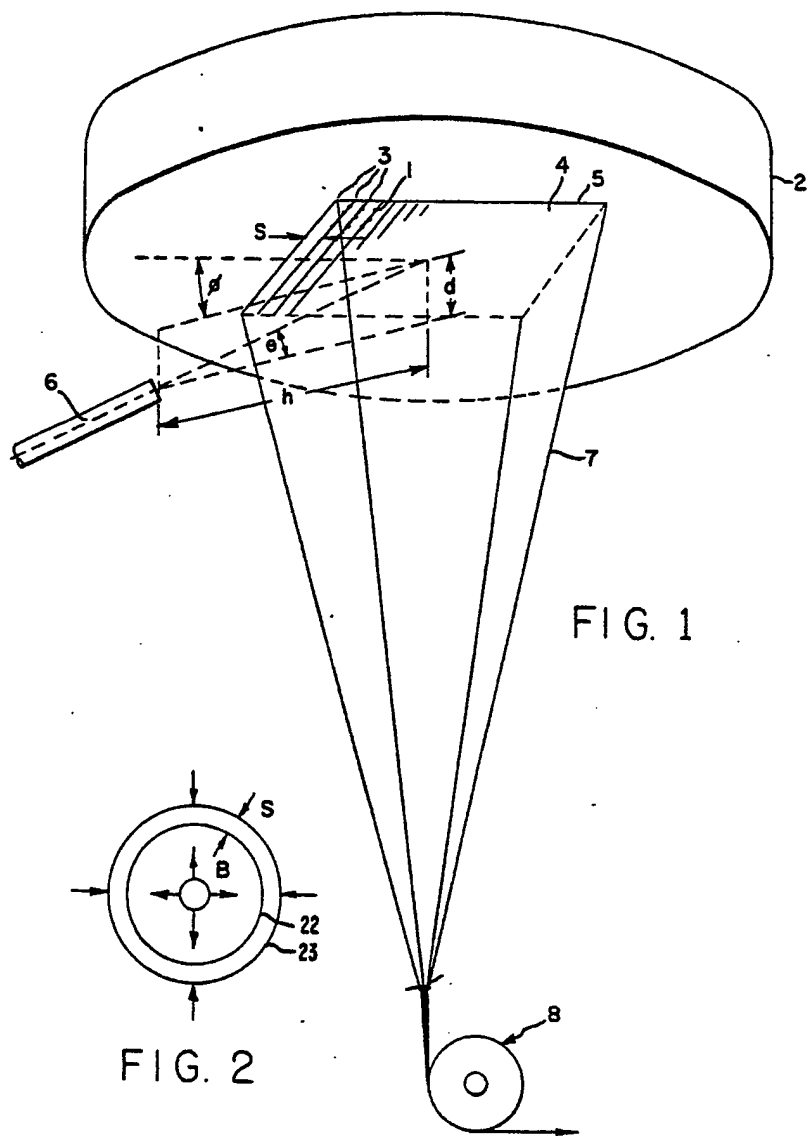
17. Filamentary materials obtained by a process claimed in any of the preceding claims, and fabrics and other articles containing them.

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FIG. 1

FIG. 2

936729   COMPLETE SPECIFICATION

2 SHEETS   *This drawing is a reproduction of
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Sheets 1 & 2

FIG. 3

FIG. 5

FIG. 6

FIG. 7

FIG. 4

FIG. 1

FIG. 8

FIG. 9

FIG. 10

936729   COMPLETE SPECIFICATION
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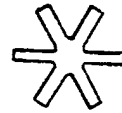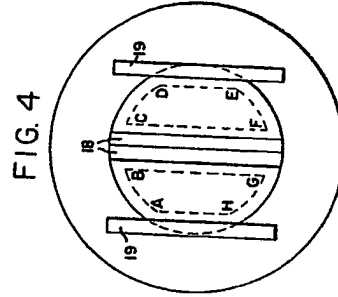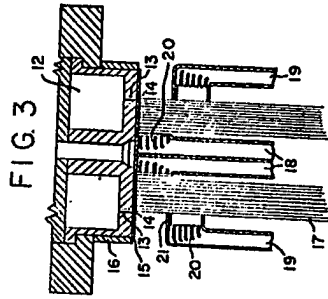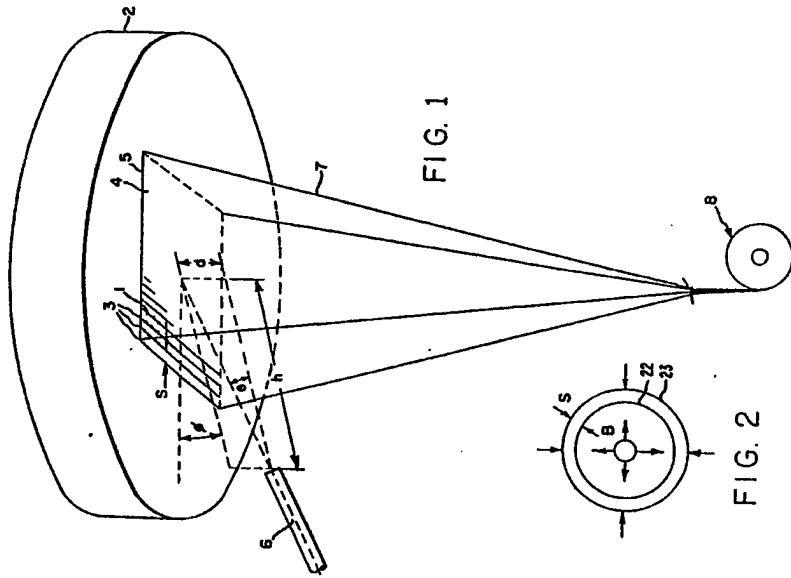the Original on a reduced scale*
Sheets 1 & 2

FIG. 5

FIG. 6

FIG. 7

FIG. 8

FIG. 9

FIG. 10

FIG. 3

FIG. 4

FIG. 1

FIG. 2